PATENT SPECIFICATION

(11) 1 454 111

1 454 111

(21) Application No. 59552/73 (22) Filed 21 Dec. 1973

(44) Complete Specification published 27 Oct. 1976

(51) INT CL² B22F 1/00 C22C 21/16 21/20

(52) Index at acceptance

C7D 8A1 8D 8K A1

C7A 71X B249 B25X B25Y B289 B309 B319 B325 B327 B329 B32Y B33X B349 B35Y B361 B363 B365 B367 B36X B389 B399 B419 B439 B459 B489 B519 B539 B549 B559 B610 B613 B616 B619 B621 B624 B627 B62X B630 B635 B661 B663 B665 B667 B669 B66X B670

(19)

(72) Inventor SAMUEL STORCHHEIM

(54) SINTERABLE POWDER COMPOSITIONS

(71) We, TOYOMENKA (AMERICA), INC., a Corporation organised and existing under the laws of the State of New York, United States of America of One World Trade Center, New York, State of New York 10048, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to aluminum powder metallurgy and, more particularly, to aluminum-copper-magnesium powder mixtures almost wholly composed of aluminum, with small amounts of copper and magnesium, which yield sintered compacts of improved mechanical properties.

It is old in the art of aluminum powder metallurgy to increase the strength of the final sintered compact by blending copper powder with the aluminum powder prior to compacting and sintering. In general, effective amounts of added copper powder have ranged from 1 to 10% by weight of the aluminum powder and have yielded sintered compacts having a typical maximum tensile strength of about 26,000 psi and an elongation of about 2% when using about 4% by weight of copper.

The copper powder for producing high strength products has been in the form of both massive and flake-like particles. The flake-shaped particles, when used, have been of the "leafing" type which, when suspended in a liquid vehicle and applied as a lacquer or paint, arrange themselves in planes parallel to the surface to which they are applied. In the case of aluminum powder metallurgy, the advantage of using leafing-type copper flake is that the surface of the flake contains a sufficient quantity of stearic acid, which produces the "leafing" property, to promote mixing of the copper with the aluminum powder and to act as a lubricant facilitating compaction of the resulting aluminum-copper powder mixture. In general, the "leafing" type copper flake powders contain 0.5 to 2% by weight of stearic acid or oleic acid on their surfaces, as described in U.S. Patent No. 3,333,950, although commercially available "leafing" copper powders generally contain 1 to 2% by weight of stearic or oleic acid. Commercially available "non-leafing" copper flake powders usually contain from 0.13 to 0.25% by weight of stearic acid added as a lubricant in the milling process required to convert massive copper particles into flakes, and when these non-leafing copper flake powders are added to aluminum powder they totally resist distribution through the aluminum powder even after 24 hours of continuous mixing.

We have now discovered that when non-leafing copper flake powder contains up to a maximum of 0.05% weight percent of a leafing-type lubricant such as stearic acid or oleic acid on its surface, it visibly mixes readily with aluminum powder and, when mixed for a minimum of one-half hour, preferably at least 1-1/2 hours, yields sintered compacts of significantly higher tensile strength and of superior surface appearance to those aluminum-copper sintered compacts obtainable heretofore. The aluminum powder further contains a conventional amount of magnesium such as to accelerate or "activate" sintering of a compact of the metal powder mixture. The mixture is essentially free from added internally lubricating amounts of any organic material.

The powder mixture of the invention can use virtually all grades of aluminum powder, the only limitation being that the finer the powder the poorer its flow rate for discharge into a die under commercial operating conditions. For example Alcoa's aluminum powder No. 1220 (9.7% minus 325 mesh Tyler), No. 120 (35.6% minus 325 mesh) and No. 123 (89% minus 325 mesh) have been used successfully with the conditional qualification, as mentioned

previously, that the coarser powders had better flow characteristics.

A small amount of magnesium in the powder mixture of the invention accelerates or "activates" sintering of the mixture by forming a relatively lower melting point eutectic with the aluminum. The amount of magnesium useful for this purpose ranges between 0.2 and 2 weight percent of the aluminum in the powder mixture. Within this range, amounts of magnesium from 0.3 to 0.6 weight percent of the aluminum are presently preferred. The magnesium can be added as magnesium metal powder or in the form of a magnesium alloy powder. When added as an alloy, it is advantageous to use as the alloying constituent one of the other components of the powder mixture, such as aluminum or copper (i.e. aluminum-magnesium and copper-magnesium alloy powders) but other alloying constituents which are advantageous for, or at least not inimical to, the desired properties of the sintered compact can be used.

The copper flake powder is used in the powder mixtures of the invention in amounts ranging from 2 to 6 weight percent of the aluminum component. Amounts of copper of at least 2% are required to impart age-hardening characteristics to sintered compacts made from the mixture, and amounts of copper in excess of 6% increase the strength and hardness of the sintered compacts at too much expense in loss of ductility. An amount of copper powder about 4 percent by weight of the aluminum powder presently appears to give a generally optimum combination of physical and mechanical properties.

The copper flake powder used in the metal powder mixtures of the invention must be of the non-leafing type but must contain the so-called "leafing" organic coating in an amount limited to a maximum of 0.05, and preferably from 0.03 to 0.05, weight percent of the copper flake. Copper flake powders are produced by flattening conventional copper particles of massive shape in the presence of a lubricating or polishing organic compound such as stearic acid, oleic acid, zinc stearate and lithium stearate. In working with copper flake powders which, as pointed out in U.S. Patent No. 3,333,950, have a density close to that of aluminum powder, we found that the "leafing" copper flake powders containing between 0.5 and 2 weight percent of the organic lubricating compound did not mix into the aluminum powder but remained as red colored striations or waves in the mixture even after mixing for 24 hours in a double cone blender. Such "leafing" copper flakes were expected, on the other hand, to blend readily with the aluminum powder because of the relatively large amount of lubricant on the copper flake surfaces, but the lubricant did not function in this manner. Further experiments with "non-leafing" copper flake powder, which as sold commercially usually has from 0.13 to 0.25% by weight of organic lubricant but in some instances can contain as little as 0.03% of the lubricant, disclosed that most of these powders similarly resisted intimate mixing with the aluminum powder even when the amount of surface organic polishing or lubricating compound was as low as 0.13% by weight of the copper. Not until there was used copper flake containing as little as 0.05% by weight of the organic material on its surface did the copper flake enter into intimate admixture with the aluminum, and at this level, i.e. at from 0.03 to 0.05% by weight of organic compound, the copper flake powder could be seen (with the naked eye) to be blended with aluminum within five minutes of blending time.

Aluminum-copper-magnesium powder mixtures of the invention, characterized by the presence of the copper in "non-leafing" flake form preferably containing from 0.03 to 0.05% by weight of organic flaking lubricant, are further characterized by the absence of any additional quantity of the same or similar conventional powder-metallurgy internal lubricants. By excluding such added lubricants, the compacted powder mixtures of the invention can be sintered in any furnace atmosphere ranging from oxidizing to neutral to reducing in nature. Thus, the compacted powder mixtures of the invention can be sintered in air, steam, smelting furnace gases containing a variety of proportions of carbon monoxide and carbon dioxide, hydrogen, nitrogen, cracked ammonia, etc.

A study of the mixing time used to obtain an intimate physical admixture of the powder mixture components has shown that mixing time has a definite effect on the mechanical properties of the aged sintered compacts made from the powder mixture. For example, a series of runs were made using a jar-rolling technique for 2,000 gram lots of a powder mixture composed of Alcoa's No. 1202 aluminum powder, 0.6% by weight of minus 325 mesh (Tyler standard) helium-reduced magnesium powder, and 4% by weight of copper flake powder containing 0.03% by weight of stearic acid. Glass jars were filled to one-half their volume and a wire screen was inserted into them to break up any agglomerates of powder during rotation of the mixtures. Mixing time was varied from one-half hour to twenty-four hours. Results obtained were as shown in Figure 1 which is

a plot of mechanical properties versus mixing time for 4"x1/2"x1/8" bars that were green pressed to 95% theoretical density and then air sintered at 1100°F. in a furnace through which they pass at a 2 1/2" per minute belt speed (four minutes at temperature) with the furnace atmosphere consisting of ambient air. As can be seen, the curve for U.T.S. rises, peaks and then starts to slowly descend. Elongations followed the same pattern but did not drop with extended mixing time, namely, twenty-four hours. A mixing time of one hour minimum appeared to be required for this type of blending apparatus while optimum mechanical property values occurred after approximately three to four hours of mixing time.

Other properties likewise varied with the blend mixing time as indicated in Figures 2 and 3. Figure 2 shows that the plot of bend-to-fracture peaks to a maximum value at six hours mixing time while the difference in sintered lengths among the 4" long bars were at a minimum at the six hours mixing time. Figure 3 shows an approximately 20% drop in growth (which occurs in air sintering) over the 4" lengths following sintering using the six hour mixing time blend.

A scale-up of the foregoing tests was made using a five cubic foot double-cone blender in which 250 pound batches of the same powder mixture were mixed for times varying from fifteen minutes through six hours. The findings from these tests were that more thorough mixing was encountered in a shorter time with the large production scale apparatus than for the small scale roller-jar combination. As a result, mechanical properties of the sintered bars rose, peaked and started to decline at an earlier mix time; that is, the decline in mechanical properties became apparent after two hours of mixing in the double-cone blender rather than after six hours in the smaller jars.

A further comparison of the advantages of the powder mix of the invention over identical mixtures except for the use of conventional copper powder appears from the following test:

Bar samples of both the aforementioned copper flake mixture of the invention (six hours mixing time), and bar samples of the same mixture except for the use of conventional copper powder, were sintered and were then immediately quenched in room temperature water upon emerging from a continuous belt sintering furnace. Bars containing the copper flake, when quenched from 550°C. and after five days of ageing, developed an U.T.S. of 34,370 psi and an elongation of 4.0%. Bars made with the conventional copper powder reached a maximum U.T.S. of 32,200 psi with a 1.8% elongation.

WHAT WE CLAIM IS:—

1. A sinterable powder composition, which composition consists substantially of a mixture of aluminum, copper and magnesium powders essentially free from added internally lubricating amounts of any organic material and containing from 2 to 6 weight percent based on aluminum of non-leafing copper flake powder, the flakes bearing on their surface no more than 0.05 weight percent of the flakes of a leafing type lubricant, and from 0.2 to 2 weight percent based on aluminum of magnesium.

2. A sinterable powder composition according to claim 1, wherein the magnesium is present in an amount of from 0.3 to 0.6 weight percent based on aluminum.

3. A sinterable powder composition according to claim 1, wherein the magnesium is present in an amount of about 0.6 weight percent based on aluminum.

4. A sinterable powder composition according to any one of the preceding claims, wherein the magnesium is provided by a magnesium-containing alloy.

5. A sinterable powder composition according to any one of the preceding claims, wherein the copper flake powder is present in an amount of about 4 weight percent based on aluminum.

6. A sinterable powder composition according to any one of the preceding claims which has been physically mixed for a continuous period of at least 1/2 hour.

7. A sinterable powder composition according to claim 6 which has been physically mixed for a continuous period of at least 1-1/2 hours.

8. A sinterable powder composition according to any one of the preceding claims, wherein the flakes of the copper powder bear on their surface from 0.03 to 0.05 weight percent of the flakes of a leafing type lubricant.

9. A sinterable powder composition according to claim 1 substantially as hereinbefore described.

Agents for the Applicants,
GALLAFENT & CO.,
Chartered Patent Agents,
8, Staple Inn,
London, WC1V 7QH.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1976
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

1454111 COMPLETE SPECIFICATION

3 SHEETS This drawing is a reproduction of the Original on a reduced scale

Sheet 1

FIG. 1.

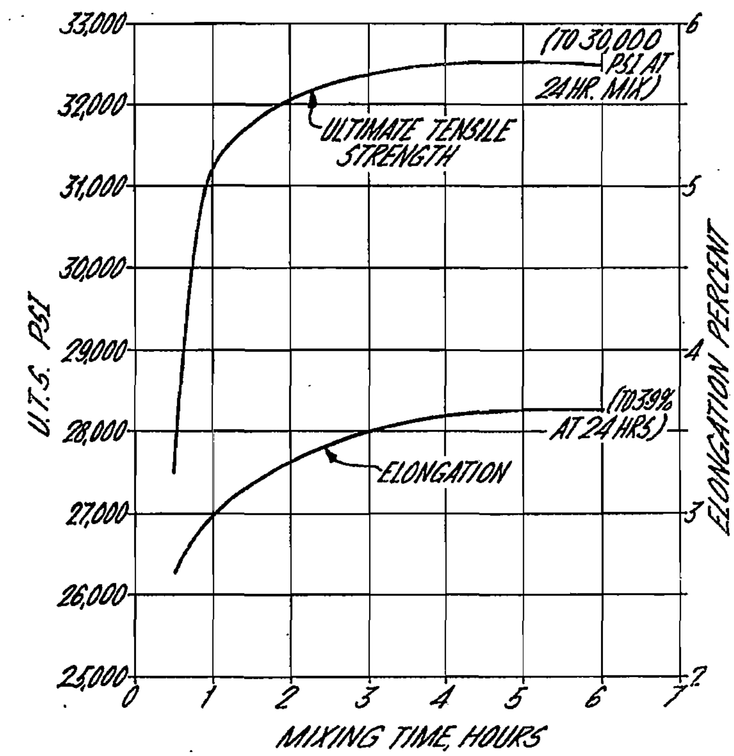


FIG. 2.

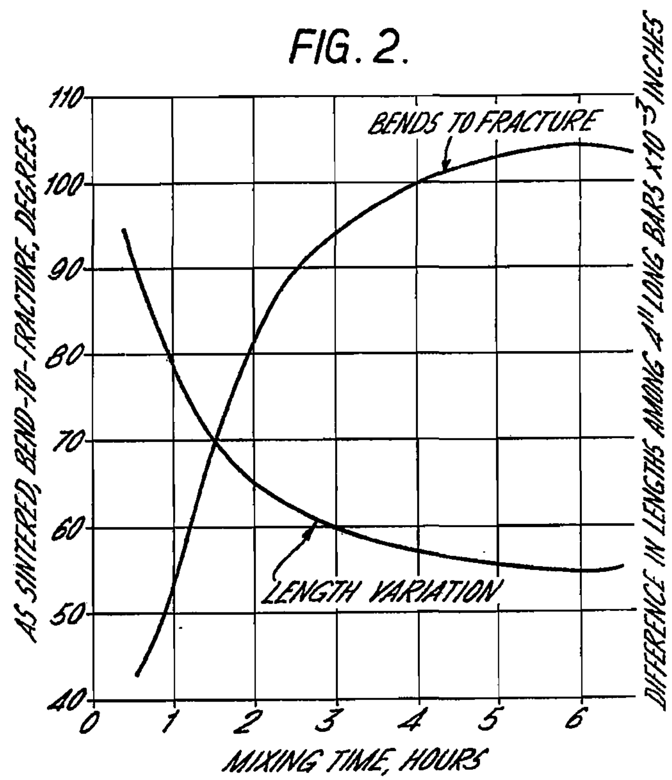
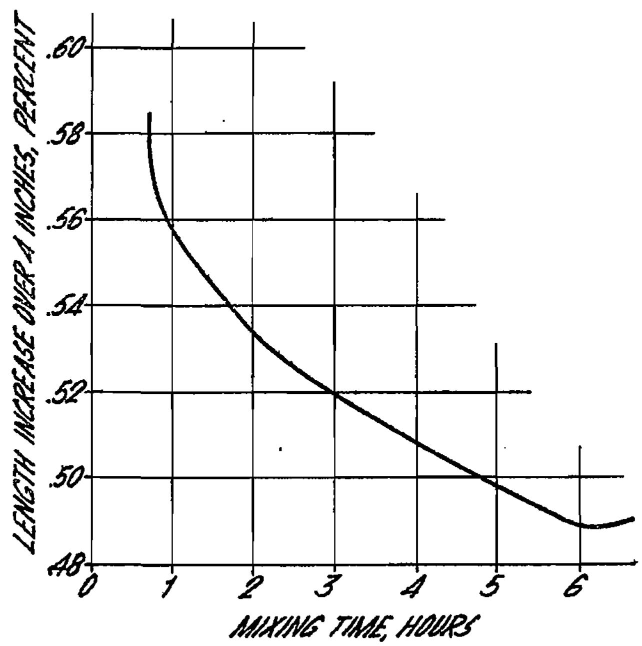

AS SINTERED BEND-TO-FRACTURE, DEGREES
DIFFERENCE IN LENGTHS AMONG 4" LONG BARS x10⁻³ INCHES
BENDS TO FRACTURE
LENGTH VARIATION
110
100
90
80
70
60
50
40
0
1
2
3
4
5
6
MIXING TIME, HOURS

FIG. 3.


LENGTH INCREASE OVER 4 INCHES, PERCENT
.60
.58
.56
.54
.52
.50
.48
0
1
2
3
4
5
6
MIXING TIME, HOURS